# Sampling & Analysis DAPP

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### **ENFORCEMENT CONFIDENTIAL**

U.S. ENVIRONMENTAL PROTECTION AGENCY TECHNICAL ENFORCEMENT SUPPORT AT HAZARDOUS WASTE SITES

> TES IV CONTRACT NO. 68-01-7351 WORK ASSIGNMENT NO. 101

CHEMETCO, INC.
HARTFORD, ILLINOIS
SITE SPECIFIC SAMPLING AND ANALYSIS
OAPP

EPA REGION V

JACOBS ENGINEERING GROUP INC. PROJECT NUMBER: 05-B101-00 SEPTEMBER 1987

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#### **PREFACE**

The objective of Metcalf & Eddy's quality assurance program is to ensure that all measurement, data gathering, and data generation activities yield data that are of adequate quality for the intended use. The key to achieving this objective is the successful implementation of a project specific Quality Assurance Project Plan (QAPP) for all such activities.

This document constitutes a site specific QAPP for the Special Study at Chemetco, Inc., Hartford, Illinois and specifically addresses Quality Assurance (QA) issues for field activities at that site. This report outlines the procedure and methods anticipated to be employed for the sampling episode by Metcalf & Eddy, Inc. and its subcontractors. This site specific QAPP supplements Metcalf & Eddy Standard Operating Procedures (SOP) by providing QA objectives, sampling procedures, sample custody protocol and analytical procedures that are unique to the sampling effort at Chemetco, Inc.

This QAPP has been prepared according to guidelines recommended in TES IV Contract 68-01-7351, and in the EPA document "Interim Guidelines and Specifications for Preparing QAPP's" QAMS-005/80 as well as the new guidelines established by EPA (August 26, 1985) based upon the type of work being done and the intended use of the data.

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#### 1.0 Project Description

The U.S. EPA is conducting investigations to determine the possible toxicity (as measured by the EP Toxicity Procedure) of waste slag from smelting operations at the Hartford, Illinois, Chemetco facility. This work is being performed under the Technical Enforcement Support (TES IV) contract to Jacobs Engineering Group, Inc. (Prime Contractor) and Metcalf & Eddy, Inc., subcontractor to the prime investigator.

The Chemetco facility consists of approximately 35 acres of land located south of Hartford, Illinois. The primary operation at the facility is the production of 99% pure copper casts from scrap metal. Through this operation lead, tin, and precious metals are also recovered.

Wastes produced from Chemetco's operations include slag from the smelting operations and emission control sludge from secondary lead smelting.

The slag waste is stored in a pile which covers approximately nine acres. The thickness of the slag in this area varies from 1 to 47 feet with an average thickness of approximately 15 feet. The size of slag "grains" ranges from dust to boulders (many 3 feet across or larger).

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Chemetco conducted representative sampling of the slag waste pile (60 samples from 20 locations at various depths) in early The analysis of the samples collected resulted in no sample exceeding the EP toxicity level for lead or cadmium. However, the analytical method which was used was reportedly not acceptable. Chemetco was informed of this problem and given a list of four labs which are approved for use under EPA's contract lab program for EP toxicity analysis. Chemetco selected a lab from this list for a re-analysis of 20 of the 60 original slag samples. EPA selected the 20 samples for re-analysis from samples stored at the Chemetco facility. Three 100 gram split samples were prepared from the stored slag samples under the supervision of an Illinois EPA representative. representative collected one split for analysis at the IEPA lab and a second split for shipment to EPA's Central Regional Lab (CRL). Chemetco shipped its split to U.S. Testing in New Jersey. The results of all analyses were forwarded along with a memo (2-27-86) from CRL explaining analytical techniques used by each lab to Metcalf & Eddy Inc. for evaluation and interpretation because the U.S. EPA has requested that the Technical Enforcement Support (TES) Contractor provide assistance in determining whether the Chemetco slag waste should be considered EP toxic.

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The objective of this study is to determine whether or not the slag waste is EP Toxic by way of lead or cadmium. Activities planned to accomplish this objective will include a site visit to re-collect representative slag samples from archived slag samples stored at Chemetco, analysis of the slag samples to determine if any sample exceeds EP Toxicity criteria for lead and cadmium, and the preparation of a written final report resolving the outstanding toxicity issues.

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#### 2.0 Project Organization and Responsibility

Figure 2.1 presents the proposed QA Organizational Chart for this project and identifies the individuals responsible for each element of the overall program. The key individual responsible for Quality Assurance is the QA Project Officer, a full-time professional reporting directly to the Corporate QA Manager who is the Senior Vice President of Metcalf & Eddy with Corporate QA responsibility. Mr. Bruce Goodwin, Technical Specialist in Metcalf & Eddy's Chemical Waste Management Group, will be QA Project Officer for this study. Analytical results are reported directly to the Project Manager who reviews the data and provides it to the QA Project Officer and the project engineer.

The project manager assigned overall responsibility for this study is Mr. Neville Chung, General Manager of Metcalf & Eddy's Eastern Region Hazardous Waste Management Group.

The Project Engineer is responsible for overseeing the implementation of the QAPP objectives while conducting the field investigations and sampling episode. For the specific sampling episode at Chemetco, Mr. Alan Ford, senior engineering technician in Metcalf & Eddy's Chemical Waste Management Group, will be charged with overseeing all on-site activities.

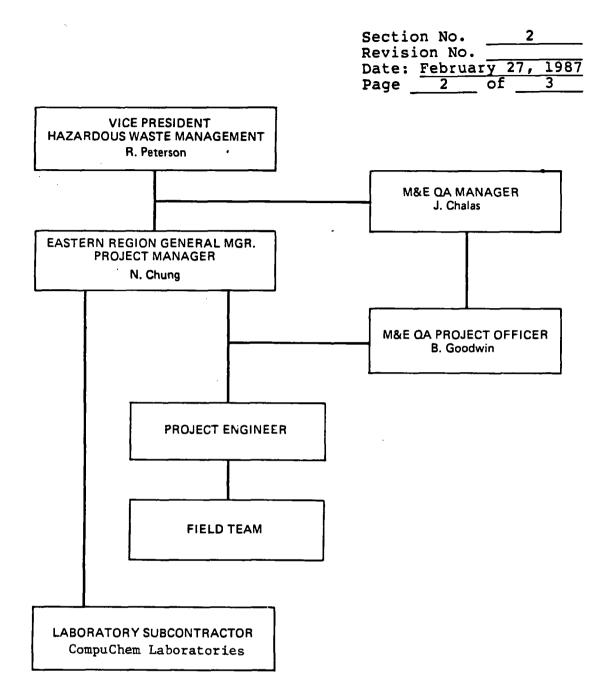


FIGURE 2.1 PROJECT ORGANIZATION

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The subcontract laboratory, which will provide analytical services in support of the sampling episode, will be CompuChem Laboratories, Inc. of Research Triangle Park, NC. CompuChem is to provide Metcalf & Eddy with EP Toxicity analysis services together with technical support in the application of the chemical data produced, and stands committed to providing chemical measurements of a quality consistent with project needs and requirements in a reasonable time while maintaining cost control. This commitment recognizes the need for data to be representative of the environmental conditions under consideration, to be valid and reliable, and suitable for making decisions that involve public health and safety, property rights, and legal liabilities. To this end, CompuChem has developed a company-wide Quality Assurance (QA) Plan and maintains an ongoing QA Program. A QA Officer is appointed by, and reports to, upper management of the Company independent of operational and budgetary concerns. CompuChem is committed to employing proper analytical methods, acquiring equipment appropriate to the methods, maintaining such equipment in good condition, securing qualified staff, and to coordinating all aspects of operation so as to produce a useful report.

Resumes of key individuals that will take part in analysis of samples generated by this study are contained in Appendix A.

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#### 3.0 Sampling

All sampling methods described in this section are standard Metcalf & Eddy SOP's based on recognized USEPA procedures as presented in "Test Methods for Evaluating Solid Waste," SW846, July 1982.

#### 3.1 Selection of Sampling Methodology

Information obtained during the initial project records review was compiled to assist with selection of sampling methodology for the smelter slag. This information included:

- U.S.E.P.A. prepared Statement Of Work (SOW)
- Case history as presented in U.S.E.P.A. memoranda of Feb. 27, 1986 and Dec. 18, 1986
- Raw analytical data as submitted by U.S. Testing Inc,
   the Illinois EPA laboratory (IEPA) and USEPA Central
   Regional Laboratory (CRL).

The selection of the actual sampling methodology utilized this information and several criteria necessary to collect valid samples from the previously archived materials.

The first criteria was that the collected samples be representative of the waste under investigation. That is, the

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collected samples must exhibit average properties of the whole waste being examined. Secondly, it was necessary to design a sampling system that would allow enough samples to be collected so that the variability of the waste material could be sufficiently characterized. By collecting representative samples, concerns over sampling accuracy will be addressed. In determining the variability of the waste, issues with regard to sampling precision can be understood. Sampling accuracy as well as sampling precision are criteria which may be of great overall importance during final review of Chemetco data. Because the initial data review revealed EP Tox values for lead and cadmium which were very close to the regulatory threshold values, highly accurate and precise sampling and analysis methodologies will be crucial to the outcome of this work.

#### 3.2 Samples to be Collected

Table 3.2 presents a list of all 60 discrete slag samples available for analysis. These samples have been retained in 10-pound sealed bags at the Chemetco facility. From these 60 discrete samples, 20 composite samples will be prepared as more particularly described below.

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TABLE 3.2
ARCHIVED SAMPLES AVAILABLE FOR ANALYSIS

1.	03P1	4.	07M1	7.	0701	10.	11X1	13.	13Q1
2.	03P2	5.	07M2	8.	0702	11.	11X2	14.	13Q2
3.	03P3	6.	07M3	9.	0703	12.	11X3	15.	13Q3
16.	21I1	19.	22XI	22.	23W1	25.	24E1	28.	24G1
17.	21I2	20.	22X2	23.	23W2	26.	24E2	29.	24G2
18.	21I3	21.	22X3	24.	23W3	27.	24E3	30.	24G3
31.	24R1	34.	25AA1	37.	25E1	40.	25L1	43.	26K1
32.	24R2	35.	25AA2	38.	25E2	41.	25L2	44.	26K2
33.	24R3	36.	25AA3	39.	25E3	42.	25L3	45.	26K3
46.	2701	49.	28K1	52.	29M1	55.	32T1	58.	34Q1
47.	2702	50.	28K2	53.	29M2	56.	32T2	59.	34Q2
48.	2703	51.	28K3	54.	29M3	57.	32T3	60.	34Q3

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By using the random number table presented below, sample aliquots were chosen for composting. Each line of the number table was read across until three previously unused sample numbers were found. The resulting composite samples to be generated are shown in Table 3.3, and Table 3.4 summarizes parameters to be measured.

 24
 06
 14
 24
 58
 58
 26
 24
 45
 42
 46

 55
 54
 29
 02
 01
 54
 08
 34
 38
 52
 14
 38
 59
 41

 49
 54
 37
 34
 11
 14
 41
 02
 12
 36
 38
 43
 36
 27

 04
 19
 48
 35
 54
 41
 47
 44
 13
 27
 50
 18
 16
 40
 02
 45
 15

 15
 52
 42
 22
 22
 38
 41
 03
 27
 15
 26
 36
 11
 33
 08

 10
 17
 05
 31
 23
 08
 07
 40
 60
 44
 16
 31
 05
 46
 41
 47

 40
 42
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#### RESULTS OF RANDOM NUMBER SELECTION

Composite #	Sample Combinations <sup>1</sup>		Composite #	Sample Combinations <sup>1</sup>			
1	24	6	14	11	47	44	13
2	58	26	45	12	50	18	16
3	42	46	55	13	40	15	22
4	54	29	2	14	3	33	10
5	1	8	34	15	17	5	31
6	38	52	59	16	23	7	60
7	41	49	37	17	51	57	9
8	11	12	36	18	21	25	28
9	43	27	4	19	20	32	56
10	19	48	35	20	30	39	53

<sup>(1)</sup> Numbers in this column refer to samples identified in Table 3.2

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TABLE 3.3

COMPOSITE SAMPLES TO BE PREPARED

Composite #	Chemetco Sample Combinations
1	23W3 + 07M3 + 13Q2
2	34Q1 + 24E2 + 26K3
3	25L3 + 2701 + 32Tl
4	29M3 + 24G2 + 03P2
1 2 3 4 5	03P1 + 0702 + 25AA1 Lab Duplicate $(1)$
	25E2 + 29M1 + 34Q2
6 7 8 9	25L2 + 28K1 + 25E1
8	11X2 + 11X3 + 25AA3
9	26Kl + 24E3 + 07Ml
10	22X1 + 2703 + 25E2 Field Duplicate $(1)$
11	2702 + 26K2 + 13Q1
12	28K2 + 21I3 + 21I1
13	25L1 + 13Q3 + 23W1
14	03P3 + 24R3 + 11X1
15	2112 + 07M2 + 24Rl Field Triplicate (1)
16	23W2 + 0701 + 34Q3
17	28K3 + 32T3 + 0703
18	22X3 + 24E1 + 24G1
19	22X2 + 24R2 + 32T2
20	24G3 + 25E3 + 29M2 Field Duplicate (1)

A systematic system was used to determine which samples would be designated for the additional Quality Control determinations. After actual observation of the samples in the field, all information will be reviewed and selection of alternate samples for QC determinations may be made because of the new information obtained.

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TABLE 3.4. PARAMETERS TO BE MEASURED

Sample Composite No.		Analysis Description
1, 2, 3, 4, 6, 7, 8, 9, 11, 12, 13, 14, 16, 17,	1.	Single aliquots collected in field.
18, 19	2.	EP Extract prepared from each composite.
	3.	
10, 20 (Field Duplicates) <sup>1</sup> , <sup>2</sup>	1.	Duplicate aliquots collected in field.
•	2.	One EP Extract prepared from each composite.
	3.	Two Pb and Cd data sets reported.
5 (Laboratory Duplicate) <sup>1, 3</sup>	1.	Single aliquots collected in field.
(Jasouanos) Japanes,	2.	Two identical composites and EP Extracts prepared from composites.
	3.	Two Pb and Cd data sets reported.
15 (Field Triplicate) <sup>1</sup> , <sup>2</sup>	1.	Triplicate aliquots collected in field.
(11010 111p110000)	2.	One EP Extract prepared from each composite.
	3.	

A systematic system was used to determine which samples would be designated for the additional Quality Control determinations. After actual observation of the samples in the field, all information will be reviewed and selection of alternate samples for QC determinations may be made because of the new information obtained.

Field duplicates and triplicates are used to provide insight into sampling and matrix variability (sample accuracy).

Laboratory duplicates are intended to indicate analytical variability (laboratory precision).

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#### 3.3 Sample Collection Methods, Archived Slag Samples

Chemetco collected samples of slag waste in early 1985. Sixty discrete samples from twenty locations collected at various depths have been stored in ten pound bags held by Chemetco, and it is from these sixty bags that 20 composite samples will be prepared for this study.

As discussed, the sixty original samples collected represent twenty sampling locations at various depths. In order to reduce the number of discrete samples submitted to the laboratory for analysis, 20 composite samples will be prepared by randomly selecting 20 sets of 3 samples which will be composited on an equal weight basis under laboratory conditions. By compositing samples collected at various depths from various locations, the resulting data can be used to represent an average concentration of cadmium and lead encountered at the Chemetco site regardless of depth or specific sample location.

The procedure to be used to prepare the twenty composites and resulting samples for analysis is presented below in a project-specific standard operating procedure (SOP).

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#### 3.3.1 Composite Sample Preparation, -- Waste Slag from Bags

#### Equipment Needed:

- 1. 72, 175 ml wide-mouth polyethylene bottles with screw-cap, tight fitting poly caps. Bottles to be prepared for use as described in Section 4.0.
- 2. Sample trier, approximately 60 cm in length (Primary sampling tool).
- 3. Sample auger, equipped with aluminum pan as described below (optional sampling tool).
- 4. Water-proof sample labels, indelible marking pen, chain of custody bottle seals and forms.
- 5. Dust tight shipping container.

#### Procedure:

- Arrange all ten-pound sample bags so that each sample label is clearly visible.
- Clean sample auger or trier as described in Section 4.2.1 drying thoroughly with lab-type wipes.
- 3. Place cleaned trier on uncontaminated surface and protect from dust.
- 4. Label sample bottles with indelible ink. Record the following information on each composite bottle:
  - a. Sample location identification.
  - Identification of each sub-sample making up composite.
  - c. Today's date
  - d. Name of person collecting the sample for composting.
  - e. Analysis requested.
  - f. Project number.

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- 5. Open bag of archived sample. Record physical appearance of material in field log book.
- 6. Grasp handle of trier and firmly plunge trier vertically downward through material to be sampled as far as possible.
- 7. Rotate trier to cut a core of sample from within the bag.
- 8. Withdraw trier containing sample and carefully transfer the material into the pre-labeled sample bottle. Close bottle.
- 9. Reclean trier. Continue collection of sub-samples as above until all 60 samples from all depths are collected.
- 10. Seal bottles and prepare chain of custody forms as well as shipping papers as outlined in Section 5.0.
- 11. Transmit samples to CompuChem with instructions to prepare 20 composite samples. The laboratory is to composite samples on a weight basis, taking equal amounts of each sub-sample identified in Table 3.3 to form the 20 specified composites for analysis.
- 12. If the archived samples are hard packed and solid, a sampling auger may be substituted for the trier. In such a case, the following procedure would be implemented.
  - Clean auger as described in Section 4.2.1.
  - b. Bore a hole through the middle of an aluminum pie pan large enough to allow the blade of the auger to pass through. The pan will be used to catch the sample brought to the surface by the auger.

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- c. Place pan against the sampling point. Auger through the hole in the pan until the desired sampling depth is reached. Back off the auger and transfer the sample in the pan and adhering to the auger to a container. Spoon out the rest of the loosened sample with a sample trier.
- d. Proceed as explained in Trier procedure above, substituting auger for trier.

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#### 4.0 Decontamination Procedures

The cleaning procedures outlined in this section are to be used by all M&E personnel to clean sampling and other field equipment as well as sample containers prior to field use. Sufficient clean equipment and sample containers should be transported to the field so that the entire investigation can be conducted without the need for cleaning equipment in the field. Since this will not always be possible when using specialized field equipment, field cleaning procedures are included to cover these special problem areas.

These procedures are the standard operating procedures (SOP) for this project; any deviation from them <u>must</u> be documented in field records and investigative reports.

#### 4.1.1 Cleaning Materials

The cleaning materials referred to throughout this section are defined in the following paragraphs.

The laboratory detergent shall be a standard brand of phosphate-free laboratory detergent such as Sparkleen® or

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Liquinox®. The use of any other detergent must be justified and documented in the field log books.

The nitric acid solution shall be made from ACS reagentgrade nitric acid and deionized water.

Tap water may be used from any municipal water treatment system. The use of an untreated or non-potable water supply is not an acceptable substitute for tap water.

Deionized water is defined as tap water that has been treated by passing it through a standard deionizing resin column. Most commercial systems utilize a 5-micron prefilter followed by a mixed bed deionization unit to produce deionized water. The deionized water should contain no heavy metals or other inorganic compounds.

The brushes used to clean equipment as outlined in the various sections of this procedure shall not be of the wire-wrapped type.

The nitric acid solution, laboratory detergent, and rinse waters used to clean equipment shall not be reused.

#### 4.1.2 Marking of Cleaned Sampling Equipment and Containers

All equipment and sample containers that are cleaned utilizing these procedures shall be labeled or marked with the date that the equipment was cleaned. Also, if there was a

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deviation from the standard cleaning procedures outlined in this section, this fact should be noted on the label.

When sample containers are cleaned and prepared, they should be cleaned in standard sized lots to facilitate the quality control procedures outlined in Section 4.2

#### 4.1.3 Marking and Segregation of Used Field Equipment

Field or sampling equipment that needs to be repaired shall be identified with a red tag. Any problems encountered with the equipment and needed repairs shall be noted on this tag. Field equipment or reusable sample containers needing cleaning or repairs shall not be stored with clean equipment, or sample containers. Field equipment and reusable sample containers that are not used during the course of this investigation may not be replaced in storage without being recleaned if these materials have been transported to the facility.

### 4.1.4 Decontamination of Equipment Used to Collect Samples of Toxic or Hazardous Waste

Equipment that is used to collect samples on this site shall be decontaminated before it is returned from the field. At a minimum, this decontamination procedure shall consist of washing with laboratory detergent and rinsing with tap water. More stringent decontamination procedures may be required, depending on the waste sampled.

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#### 4.1.5 Proper Disposal of Cleaning Materials

The nitric acid solution and any other liquids used to rinse sampling equipment and containers shall be collected and disposed of through an approved hazardous waste disposal contract. These procedures apply whether the cleaning operations take place in a laboratory or in the field.

## 4.1.6 Use of Safety Procedures to be Utilized During Cleaning Operations

The materials used to implement the cleaning procedures outlined in this SOP can be dangerous if improperly handled. Caution must be exercised by all personnel and all applicable safety procedures shall be followed. At a minimum, the following precautions shall be taken in the lab and in the field during these cleaning operations:

- Safety glasses with splash shields or goggles, neoprene gloves, and a neoprene laboratory apron will be worn during all cleaning operations.
- 2. All acid rinsing operations will be conducted under a fume hood or in the open (never in a closed room).
- 3. No eating, smoking, drinking, chewing, or any hand to mouth contact shall be permitted during cleaning operations.

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#### 4.1.7 Storage of Field Equipment and Sample Containers

All field equipment and sample containers shall be stored in a contaminant free environment after being cleaned using the procedures outlined in this section.

- 4.2 SPECIFIC QUALITY CONTROL PROCEDURES FOR CLEANING OPERATIONS
- 4.2.1 CLEANING PROCEDURES FOR STAINLESS STEEL OR METAL TRIERS, AGUERS AND SIMILAR EQUIPMENT USED FOR THE COLLECTION OF SAMPLES FOR METALS ANALYSES\*
  - Wash equipment thoroughly with laboratory detergent and hot water using a brush to remove any particulate matter or surface film.
  - Rinse equipment thoroughly with hot tap water.
  - Rinse equipment thoroughly with deionized water.
  - 4. Dry equipment thoroughly using clean lint-free disposable wipers.

<sup>\* -</sup> When this sampling equipment is used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide grade acetone or hexane to remove the materials before proceeding with Step 1. In extreme cases, when equipment is painted, badly rusted, or coated with materials that are difficult to remove, it may be necessary to steam clean, wire brush, or sandblast equipment before proceeding with Step 1. Any stainless steel or other sampling equipment that cannot be cleaned using these procedures should be discarded.

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#### 4.2.2 MISCELLANEOUS EQUIPMENT CLEANING PROCEDURES

#### 4.2.3 <u>Ice Chests and Shipping Containers</u>

All ice chests and reusable containers will be washed with laboratory detergent (interior and exterior) and rinsed with tap water and air dried before storage. In the event that an ice chest becomes severely contaminated with concentrated waste or other toxic material, it shall be cleaned as thoroughly as possible and disposed of properly.

#### 4.2.4 Vehicles

All vehicles utilized by M&E should be washed at the conclusion of each field trip. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. When vehicles are used in conjunction with hazardous waste site inspections, or on studies where toxic materials are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of such investigations. It shall be the responsibility of the Project Engineer and/or field investigators to see that this procedure is followed.

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#### 4.3 PREPARATION OF SAMPLE CONTAINERS

#### 4.3.1 General

No sample container will ever be reused. All sample containers will be stored in their original packing cartons. When packages of uncapped sample containers are opened, they will be placed in new plastic garbage bags and sealed to prevent contamination during storage. Specific precleaning instructions for sample containers are given in the following sections.

### 4.3.2 <u>175 ml Polyethylene Bottles for EP Toxicity Slag Samples.</u>

- 1. Wash Polyethylene bottles and caps in hot water with laboratory detergent.
- 2. Rinse both with at least 10% nitric acid solution.
- 3. Rinse three times with deionized water.
- 4. Invert bottles and dry in contaminant free environment.
- 5. Cap bottles.
- 6. Store in contaminant free area.

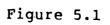
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#### 5.0 Sample Handling and Chain of Custody

An important consideration for environmental measurement data is the ability to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt and laboratory custody until disposal must be documented to accomplish this. Documentation is accomplished through a chain of custody record that records each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- . In a person's actual possession.
- . In view after being in physical possession.
- Locked so that no one can tamper with it after having been in physical custody.
- . In a secured area, restricted to authorized personnel.

Sample custody will be initiated by Metcalf & Eddy field personnel upon collection of samples. Documents specifically prepared for such purposes will be used for recording pertinent information about the types and numbers of samples collected and shipped for analysis. An example chain of custody form is included as figure 5.1. The samples collected will first be brought to an on-site location for batching and paperwork



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checks. Labels and log information are checked to be sure there is no error in identification. Samples are packaged to prevent breakage or leakage, indicate tampering during transit, and labeled according to DOT regulations for transport by air as laboratory samples. These procedures are outlined in Section 8.0. Copies of forms will be maintained for the project record. Storage of samples by the laboratory will be under conditions specified for the analyses to be performed. Samples used for analysis will be held for 60 days following report of the data before disposal. Any archived samples will be stored until the end of the project, or shipped to another lab (for reanalysis if necessary). Samples are handled by the laboratory as described in the following paragraphs.

Any samples that are later designated to be sent to a second laboratory for independent confirmation of analytical results will contain copies of the chain of custody documentation and are to be shipped according to DOT regulations. The independent lab will be responsible for proper disposition of the samples received.

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#### 5.1 Chain of Custody Record Form

Figure 5.1 is an example of the chain of custody form to be used by Metcalf & Eddy personnel in collecting and shipping samples associated with this study.

The chain of custody form shall be signed by each individual who has had the samples in their possession. Preparation of the chain of custody form shall be as follows:

- . The chain of custody record shall be initiated for every sample by the person collecting the sample. Every sample shall be assigned a unique identification number that is entered on the chain of custody form. Samples can be grouped for shipment using a single form.
- . The record shall be completed in the field using indelible ink to indicate project, sampling team, etc.
- . The person transporting the samples for shipment shall sign the record form as Transported By \_\_\_\_\_.
- Because the samples are to be shipped to the laboratory by commercial carrier, the chain of custody form shall be sealed in a watertight envelope, placed in the shipping container, and the shipping container sealed prior to being given to the carrier.
- . The commercial carrier's airbill shall serve as an extension of the chain of custody record between the final field custodian and receipt in the laboratory.
- . Upon receipt in the laboratory, the Quality Control Coordinator, or representative, shall open the chain of custody record, and sign and date the record. Any discrepancies shall be noted on the chain of custody form.

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- . If discrepencies occur, the samples in question shall be segregated from normal sample storage and the field personnel immediately notified.
- . Chain of custody records shall be maintained with the specific project files, becoming part of the permanent project documentation.

#### 5.2 Field Collection and Shipment

In addition to initiating the chain of custody form, field personnel are responsible for uniquely identifying (required on the chain of custody form) and labeling samples, providing proper field documentation, and packaging samples to preclude breakage during shipment.

Every sample shall be labeled using indelible ink to include the following:

- . Project number.
- . Unique sample number.
- . Sample description (such as borehole and depth, or grid coordinates).
- . Date of compositing.
- . Person obtaining the sample.
- Analysis requested.

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Samples must be placed in containers compatible with the intended analysis. Requirements for the specified analytical parameters with respect to the type of container, preservation method, and maximum holding time between collection and analysis have been presented in Section 7.0.

Shipping containers are to be sealed prior to shipment, both during direct transport via field personnel as well as when commercial carrier is used.

As soon as field personnel are ready to transport samples from the field to the laboratory, they shall notify the laboratory by telephone of the shipment. The estimated time of arrival at the laboratory should be given.

## 5.3 Laboratory Sample Receipt

Upon sample receipt, the QC Coordinator, sample custodian or his designee shall:

Examine all samples and determine if sample integrity has been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were

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affected. Any samples affected shall also be considered damaged. It will be noted on the chain of custody record that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible that samples were damaged and that they must be resampled, or the testing program changed.

- Compare samples received against those listed on the chain of custody.
- . Verify that sample holding times have not been exceeded.
- . Sign and date the chain of custody form and attach the airbill to the chain of custody.
- List the samples in the laboratory sample master log-in book which contains the following information:
  - Project identification number
  - Sample numbers
  - Type of samples
  - Date received in laboratory
- . Notify the Laboratory Manager of sample arrival.
- . Place the completed chain of custody records in the project file.

#### 5.4 Laboratory Storage of Samples.

The primary considerations for sample storage are:

- . Maintenance of sample integrity. Typically samples will be stored at four degrees celcius.
- Extracting and analyzing samples within the prescribed holding time for the parameters of interest.

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Placement of samples in the proper storage environment is the responsibility of the QC Coordinator, who should notify the Laboratory Manager or his designated representative, if there are any samples which must be analyzed immediately because of holding-time requirements.

## 5.5 Initiation of Testing Program.

The Laboratory Manager is responsible for prioritizing samples on the basis of holding time and required reporting time into the laboratory sample stream.

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### 6.0 Sampling Program Organization

The following table 6.1 outlines the expected time table and chronology of events that will occur during the Chemetco Study and field sampling episode.

TABLE 6.1. CHRONOLOGY OF EVENTS CHEMETCO SITE

Date Window	Event
02/04/87 - 02/19/87	Work Plan Submittal
02/19/87 - 03/04/87	Sampling Plan Submittal
08/24/87 - 08/31/87	QAPP Submittal, Second Revision
08/31/87 - 09/15/87	EPA Approval of Revised Sampling and QAPP
09/16/87 - 09/30/87	Collection of Samples
10/20/87 - 11/15/87	Draft Report Submittal
11/16/87 - 11/30/87	EPA Approval of Draft Report
12/01/87 - 12/15/87	Final Report Delivered

This schedule assumes EPA review of the revised Sampling Plan and QAPP will take no longer than 15 days, and, that EPA review of the Draft report can be accomplished in 15 days as well.

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## 7.0 Sample Containers and Preservation Requirements

Table 7.1 presents container requirements, preservation specifications and laboratory holding times to be adhered to during all sampling and analysis activities associated with this project.

TABLE 7.1.

#### SLAG SAMPLES

Parameter Containe	<sub>r</sub> (1)	Preservation (2)	Holding Time (3)
EP Toxicity, selected metals	3 x 175 ml, P, unlined P lids	None	Extract ASAP
Metals, (Cd, Pb)	In EP extract	Cool, 4°C	6 months

- P, Polyethylene with unlined polyethylene cap.
- Slag samples are not chemically preserved.
- 3. Samples should be analyzed as soon as possible after collection. Samples may be held for longer periods only if the laboratory has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional EPA Administrator. Some samples may not be stable for the maximum time period given in the table. A laboratory is obligated to hold the samples for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

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## 8.0 Sample Packaging and Shipping

In order to ensure safe, secure delivery of all collected samples to the analytical laboratory involved, the following packaging, labeling and shipping procedures have been prepared for this project. All procedures presented below are written to comply with applicable DOT regulations for transportation by surface and air.

Unless information collected during on-site activities indicates otherwise, all samples collected at the Chemetco site will be treated as non-hazardous.

Because of the expected non-hazardous nature of the collected samples, packaging and shipping criteria have been designed only to maintain chain-of-custody protocol as well as prevent breakage of the sample containers.

## 8.1 Packaging and Shipping - Field Procedure

- Place a signed, dated, chain of custody seal on the bottles in such a way that no bottles may be opened without breaking the seal.
- 2. Wrap properly labeled and secured sample bottles with two thicknesses of plastic bubble wrap. Place the wrapped containers into a water-tight zip lock bag. Seal and label the outside of the bag with the sample number or other field assigned identifier.

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- 3. Put a layer of cushioning material (e.g., styrofoam board) in the bottom of the all metal, watertight shipping containers.
- 4. Place sample bottles, tops up, in the shipper.
- 5. Use pieces of rigid styrofoam as necessary to ensure that there will be no shifting of bottles during transport.
- 6. Seal chain of custody forms in a zip-lock plastic bag and tape securely to the inside of the cooler lid.
- 7. Close and lock or latch the shippers. Seal the space between the container body and lid with waterproof tape.
- 8. Apply several wraps of pre-printed chain of custody tape around the shipping containers perpendicular to the seal to assure that the lid will remain closed if the latch is accidentally released or damaged and to prevent tampering during shipment.
- 9. If the shipping container used is an all metal picnic cooler, tape the drain plug closed so it will not open.
- 10. Place a completed Federal Express Airbill on the lid of the cooler including name, address and phone number of the receiving laboratory and the return address and phone number of the shipper.

Samples will be shipped to the laboratory via Federal Express Priority 1.

Samples will be shipped from the Federal Express Office which is closest to the site or at the closest airport directly to the laboratory. One of the M&E field team members will deliver the properly labeled sample packages to the Federal Express Office or the appropriate airport. Office hours are

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normally 8:00 am to 6:00 pm, Monday through Friday and 8:00 am to 5:00 pm on Saturday.

For each shipment to the laboratory, a Federal Express air bill must be properly completed. An example of a properly prepared air bill to be used to ship samples to the laboratory is shown as Figure 8.1.

Figure 8.1

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## 9.0 Analysis Parameters, Analytical Methods

Table 9.0 summarizes parameters to be measured as well as specific analytical methods to be used during the analysis of the slag samples collected at the Chemetco site.

## 9.1 Data Quality Objectives for Critical Measurements

Precision and accuracy goals for sampling and analysis depend upon the types of samples and analyses to be performed. For the sampling program at Chemetco, aliquots of slag will be collected from archived sample material.

Because the aliquots to be collected as part of this study will be prepared from archived sample materials previously collected by Chemetco, the accuracy as well as representativeness of the samples collected cannot be completely understood.

Quality assurance objectives in terms of precision, accuracy, and completeness are summarized in Table 9.1.

The usefulness of sampling and analysis data is contingent upon meeting criteria for representativeness and comparability. Wherever possible, only reference methods and standard sampling procedures will be followed. The main QA objective is that all measurements be representative of the archived slag samples and,

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TABLE 9.0. PARAMETERS TO BE MEASURED/METHOD REFERENCES CHEMETCO SLAG

1

Parameter Description	Method No.	Title	Method Reference
EP Toxicity Extraction	1310	Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test	Test Methods for Evaluating Solid Waste, SW-846, 2nd ed., U.S. EPA, July 1982.
Metals on EP Extract			
Cadmium	7130	Cadmium (Atomic Absorption, Direct Aspiration Method)	Test Methods for Evaluating Solid Waste, SW-846, 2nd ed., U.S. EPA, July 1982.
Lead	7420	Lead (Atomic Absorption, Direct Aspiration Method)	Test Methods for Evaluating Solid Waste, SW-846, 2nd ed., U.S. EPA, July 1982.
Digestion	3010	Acid Digestion for Flame Atomic Absorption Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 2nd ed., U.S. EPA, July 1982.

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TABLE 9.1. PRECISION, ACCURACY AND COMPLETENESS OBJECTIVES CHEMETCO SLAG EP TOXICITY ANALYSES

Parameter	Method No.	Title	Precision(1)	Accuracy <sup>(2)</sup>	Complete- ness
Metals on EP Extract					
Cadmium	7130	Cadmium (Atomic Absorption, Direct Aspira- tion Method)	<u>+</u> 10%	95%	100%
Lead	7420	Lead (Atomic Absorption, Direct Aspira- tion Method)	<u>+</u> 10%	95%	100%

Expressed as Relative Percent Difference (RPD) of duplicate measurements made on aqueous calibration standards run during analysis of project samples.

<sup>2.</sup> Expressed as percent recovery of aqueous standard material.

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that all data resulting from the analytical activities be comparable. The use of accepted, published sub-sampling and analysis methods as well as standard reporting units will aid in ensuring the comparability of the data.

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## 10.0 Data Reduction, Validation and Reporting

Data reduction, validation and reporting responsibilities will be assigned to the key individuals given in Section 2. Consistent data quality for this program will be obtained by the application of a standard data analysis and validation process. Critical review of data is designed to isolate spurious values. Data will be reviewed at a minimum by the analyst, his/her supervisor, and the QA Project Officer. Statistical tests will be applied to data to assess determinate and indeterminate errors. Determinate errors will usually be identified through the use of spikes, control charts and analysis of differing sample sizes. Indeterminate errors will be estimated in this program through statistical calculations, duplicate analyses as well as control charts.

#### Field Date Quality Reviews

<u>Obj</u>	ective	Action	Responsible <u>Person</u>
1.	Sample and field monitoring information conforms to conditions and schedule in Section 3	Review of labeled samples and in- process samples using sample inventory records	Field Sampling team
2.	Verify sample completeness	Daily count of in- complete items	Field Sampling team

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3.	Verify completeness of field log books	Review Daily	Field Team Leader
4.	All data forms are properly completed	Review and check off during each sample collection	Field Team Leader
5.	All field generated QC samples collected as required	Review require- ments and confirm	Field Team Leader

# Laboratory Data Quality Reviews

Objective		Action	Responsible Person
1.	Verify incoming data and sample complete- ness	Daily count of number and nature of samples received versus number and nature of entries made in log. Mark "verified" on log.	Sample Custodian
2.	Verify all data forms completed	Review and check off during each analysis. Forms provided by supervisor with non-required entries marked out	Analyst
3.	Manual data re- duction procedures	Daily review of calculated values against raw data values	Analyst

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4.	Verify completeness of laboratory note-books/bench sheets	Review Weekly	Laboratory Supervisor/ Manager
5.	Verify calibration criteria	Calibration cri- teria in method reviewed and test calibration accep- tance recorded.	Analyst
6.	Verify repeatability and accuracy	Record values of replicate analyses and Control samples	Analyst

## Engineering Data Quality Reviews

<u>Objective</u>		Action	Person Person
1.	Assure completeness of field and lab data	Compare field and lab data forms against data list at each use and check off	Project Engineer
2.	Assure compara- bility of units	Review units re- ported for con- sistency in cal- culations at each use and check off.	Project Engineer
3.	Examine engineering validity of data	Review parameter extremes and transients versus expected data trends. Document data excluded on this basis	Project Engineer & Quality Assurance Project Officer

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4. Examination of statistical data

Apply tests to data groupings to be used. Record data and test results.

Project Engineer & Quality Assurance Project Officer

A final data review is also performed on a spot check basis by the Project Engineer, Field Team Leader, and Subcontract Laboratory Manager.

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#### 11.0 Quality Control Checks

All analyses performed in support of this program will be done using standardized laboratory procedures. The QC program developed by Metcalf & Eddy will make use of QC samples which are "blind" to the laboratory. Calibration check samples, method blanks, and replicate aliquot analyses (both duplicates and triplicates), are QC sample types planned for this study.

Standard calibration solutions are prepared by adding known quantities of independently prepared stock materials to deionized water. The standard solutions are used to establish instrument calibration as well as to demonstrate that an instrument or procedure is in Control before analysis of samples begins. The analyst records the standard results in the instrument logbook and the expected result must be within Control limits before sample analysis begins.

Unknown or "blind" QC standard samples may be inserted in the sample batch in a solution not recognizable to the analyst to enable an estimation of the accuracy of the analytical procedure. These "blind" QC samples are prepared and distributed to the analysts by the laboratory. The analyst does not know which sample is a QC standard, or its true value.

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A continuing check sample is one of the working calibration standards that is periodically re-analyzed and the subsequent values used to demonstrate that the original calibration is still valid.

A method or reagent blank consists of deionized water carried through the entire preparation and analysis procedure. Analytical results are corrected for the method blank values before they are reported.

Analysis of "blind" replicate aliquots (duplicates and triplicates) of actual samples will be done to enable estimation of the precision of the analytical procedure. These "blind" replicates are analyzed in addition to any replicates prescribed by the analytical procedure.

The method of standard addition (MSA) will be used during the metal analyses to indicate how the sample matrix effects the recovery of analytes. The results can be used to monitor overall laboratory performance by calculating percent recoveries. Percent recoveries are reported and discussed with the analytical data. Criteria used to interpret the results of the metal analyses will be determined after the actual sample results are examined. Field generated duplicates and triplicates will be collected and submitted "blind" to the laboratory. Such samples are intended to indicate sampling and matrix variability.

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Laboratory duplicates are samples which are divided into two or more parts after collection. Each duplicate aliquot will be sent to the CompuChem laboratory for independent analysis of the same parameters.

Laboratory generated blanks, duplicates, and standards are analyzed alongside samples to provide continuous quality control during the determination of trace constituents. The blanks are analyzed to provide data on possible carry-over contamination of samples by the extraction or digestion process and also provide background concentration levels in the reagents used during sample preparation and analysis. The duplicate analyses provide laboratory precision data while the certified standards provide a measure of accuracy.

#### 11.1 Purity of Reagents

Chemical reagents, solvents and gases are available in a wide variety of grades of purity, ranging from technical grade to various ultrapure grades. The purity of these materials required by this project varies with the type of analysis. The parameter being measured and the sensitivity and specificity of the detection system

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have been important factors in determining the purity of the reagents required. For these inorganic analyses, analytical reagent grade is satisfactory. In methods where the purity of reagents is not specified, analytical reagent grade will be used. Reagents of lesser purity than that specified by the method shall not be used. The labels on the container will be checked and the contents examined to verify that the purity of the reagents meets the needs of the particular method involved.

Reagents will be prepared and standardized with the utmost of care and technique against reliable primary standards. They must be restandardized or prepared fresh as often as required by their stability. Stock and working standard solutions will be checked regularly for signs of deterioration; e.g., discoloration, formation of precipitates, and change of concentration. Standard solutions will be properly labeled as to metal concentration, solvent, date, and preparer.

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Primary standards shall be obtained from a reliable source, pretreated (e.g., dried, under specified conditions), accurately prepared in calibrated Class A volumetric glassware and stored in containers that will not alter the reagent. A large number of primary standards are available from the National Bureau of Standards (NBS) and may be used to prepare standards and check samples during this project.

## 11.2 Calibration Procedures and Frequency

Detailed calibration procedures for analytical equipment required for this project are provided in the individual analytical methods referenced in other sections. This section provides general calibration procedures for the analytical instrumentation anticipated to meet various project requirements.

#### ATOMIC ABSORPTION SPECTROPHOTOMETERS

Calibration Standards

Prepare standards daily from the 1000 ppm stock solution. The 1000 ppm stock solution is normally prepared from the high purity metal or a salt; if a salt is used, it must be dried at 105 deg C for one hour unless otherwise specified. The working standards are verified daily by analyzing against NBS SRM 1643a, Trace Elements in Water, or a sample prepared from an EPA EMSL Trace Metals concentrate.

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#### Calibration Procedure

- 1. Set the lamp current and allow the lamp to warm up; warm-up time for hollow cathode lamps is 15 minutes; 45 minutes should be allowed for electrodeless discharge lamps.
- Set wavelength and slit width as recommended in the EPA methods manual; align the lamp or furnace; optimize all parameters.
- 3. Analyze a blank and a minimum of three calibration standards which cover the linear range of the element.
- 4. Analyze a quality Control sample prepared from an EPA Trace Metals concentrate; if the reported value is within five percent of the expected value, analysis may proceed.
- 5. During the analysis, run a blank and a standard after every seven to ten samples to ensure that no drift has occurred.
- 6. Document standard preparation in the analyst's notebook. Enter all required information in the instrument logbook and sign and date the entry.
- 7. The method of standard additions is presented in Appendix A-4.

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### 12.0 Internal Quality Assurance Procedures

As applied to chemical analyses performed during this project, Quality Assurance is the demonstration and documentation of data quality. These procedures include the recording of all quality control activities, and the assessment of analytical performance by analysis of internal and external control and audit samples as discussed in Section 11.0.

#### 12.1 Calculation of Data Quality Indicators

The quality control activities undertaken during this project will include activities designed to assure that measurement systems as well as activities specific to the given site are under control.

The quality control activities consist principally of the evaluation of data obtained from the following sample categories:

(a) calibration standards, (b) working standards, (c) LCS (d) field replicates (e) field triplicates (f) field samples (g) laboratory duplicates (h) laboratory spikes (i) laboratory method blanks,

(j) (optional) laboratory split samples. The frequency of analysis of each QC sample type is typically 10%. Procedures used to evaluate this data will include calculation of arithmetic means, standard deviations, relative percent differences for duplicate samples and comparison of differences between standards of spiked and experimentally determined values expressed as percent recovery.

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As noted in preceding sections, these data will be summarized in quality control tables or charts for each measurement parameter which will be reviewed and updated regularly.

Project-specific data evaluation procedures are dependent upon the number of field samples collected. For the most part, it is anticipated that the statistical procedures used for this work will be simple and straightforward, including, for example, calculation of limits of detection, limits of quantification, confidence intervals, and evaluation by least squares linear regression. In all cases, these procedures will be taken from EPA documents and manuals appropriate to the media under investigation. Overall guidance will be obtained from the EPA document "Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements" issued on March 30, 1984 as Chapter 5 to the EPA Quality Assurance Manual.

Equations for routing statistical measures to be used in this project are presented below.

<u>Precision</u>. Precision will be determined by the analysis of replicate samples and will be expressed as the relative percent difference, which is determined according to the following equation:

$$RPD = \frac{Range}{Mean} \times 100$$

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where: RPD

range mean = relative percent difference
= maximum value - minimum value

= average of duplicate values

When triplicate measurements have been made, precision will be calculated as the standard deviation, s, which is determined according to the following equation:

$$S = \sqrt{\frac{\sum_{i=1}^{n} x_{i}^{2} - (\sum_{i=1}^{n} x_{i})^{2}/n}{\sum_{i=1}^{n} x_{i}^{2} - (\sum_{i=1}^{n} x_{i})^{2}/n}}$$

where S = standard deviation

X. = individual measurement result

 $N^1$  = number of measurements

Relative standard deviation will be reported, and is to be calculated as follows:

$$RSD = 100 - \frac{S}{\overline{X}}$$

where RSD = relative standard deviation, expressed in percent

S = standard deviation

X = arithmetic mean of replicate measurement.

Accuracy. Accuracy will be estimated from the analysis of "blind" QC samples whose true values are known to the QA Project Officer. Accuracy will be expressed as percent recovery or as relative error. The formulas to calculate these values are:

Percent Recovery = 100 Measured Value
True Value

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Completeness. Completeness will be reported as the percentage of all measurements made that generate results judged to be valid according to project criteria compared to the total number of measurements made. The following formula will be used to estimate completeness:

$$C = 100 \frac{V}{T}$$

where C = percent completeness

V = number of measurements judged valid

T = total number of measurements

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#### 13.0 Performance and System Audits

An audit is a systematic check to determine the quality of operation of some function or activity. There are two basic types of audits: (1) laboratory performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in the measurement system; or (2) system audits of a qualitative nature that consist of review to determine conformance with quality assurance/control procedures in all laboratory, field sampling and chemical analysis activities.

## Field Sampling and Chemical Analysis Systems Audits

The QA Project Officer may schedule audits of field activities to evaluate the execution of sample identification, sample control, chain-of-custody procedures, field documentation, instrument calibration and field measurement and sampling operations. The evaluation is based on the extent to which the applicable standard operating procedures are being followed.

Field documents pertaining to sample identification and control will be examined for completeness and accuracy. Field log books, field data forms and chain of custody forms will be reviewed to see that all entries are dated and signed and that the contents are legible, written in ink, and contain accurate and inclusive documentation of project activities. Because the log book, field

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data forms and chain of custody forms provide the basis for reports written later, they will contain only facts and observations.

Language will be objective, factual, and free of personal interpretations or other terminology that might prove inappropriate.

The auditor will also check to see that chain-of-custody procedures are being followed and that samples are being kept in custody at all times and are secured in a manner to prevent tampering.

Sampling operations will be evaluated to determine if they are performed as stated in the project plan or as directed by the project manager. The auditor will check to determine that the appropriate number of samples are being collected, samples are placed in proper containers, and proper preservation, packaging and shipment protocols are being followed.

## Laboratory Systems Audit

A laboratory systems audit may be performed to assure that subcontractor laboratories are maintaining the necessary minimum levels of instrumentation and levels of experience of personnel, and that laboratory quality assurance/control procedures are in conformance with the requirements of the QAPP.

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## 14.0 Preventive Maintenance

In all of the Metcalf & Eddy subcontractor laboratories, preventive maintenance includes attention to glassware, water supply, reagents and analytical balances as well as more complex instrumentation. Table 14.1 summarizes the preventive maintenance procedures for major analytical instrumentation; also listed in the table are the spare parts normally kept in inventory to minimize instrument down time.

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TABLE 14.1. MAINTENANCE PROCEDURES AND SCHEDULE FOR MAJOR INSTRUMENTATION

Instrumentation	Maintenance Procedure/Schedule	Spare Parts
Atomic Absorption	1. Clean optical surfaces as needed.	1. Graphite tubes.
Spectrophotometers	<ol><li>Condition graphite tube before starting analysis.</li></ol>	<ol><li>Graphite contact rings</li></ol>
w/Graphite Furnace	<ol><li>Check condition of graphite contact rings weekly;</li></ol>	
<del>-</del>	replace if pitted or worn.	
	4. Clean furnace windows as needed.	

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### 15.0 Corrective Action Procedures

Corrective action procedures for this program will be initiated by the analytical personnel and their supervisors directly involved with implementing the procedures presented in the QAPP. Quality control charts for daily instrument calibration and replicate analyses will be utilized to indicate the necessity for corrective action. Control charts will be established for each procedure indicating upper and lower limits of three standard deviations as the acceptability ranges. Warning ranges are established at two standard deviations. At the point when the control charts show a determination outside the warning ranges, investigation as to the cause will be initiated. Any of the following events that occurs on the quality control chart will trigger corrective action:

- Two consecutive determinations fall outside the upper or lower control limits.
- Runs up--(seven consecutive determinations increasing in value)--or runs down (seven consecutive determinations decreasing in value).
- Three consecutive values fall above or below the warning limits (two standard deviations).

Corrective actions will also be initiated as a result of other QA activities which include performance audits, systems audits, and laboratory comparison studies.

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The corrective action relative to the control charts is related more to precision than to accuracy. These charts give clues as to when some factor, generally of a procedural nature, is causing the results to drift or when an unexpected difference beyond the control limits occurs. Data within the upper and lower control limits of the control charts are well within the precision, accuracy, and completeness criteria.

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## 16.0 Quality Control Reports to Management

All new and ongoing quality assurance activities will be summarized in a written report prepared by the Quality Assurance Project Officer and included with the laboratory test reports prepared for the sampling episode. In the final project report a separate QA Section will summarize the data quality information presented in the individual test reports.

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#### 17.0 REFERENCES

- 1. Quality Assurance Handbook for Air Pollution Measurement
  Systems. Volume I Principles. EPA-600/9-76-995, March 1976.
- 2. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II Ambient Air Specific Methods. EPA-600/4-77-027a, May 1977.
- 3. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume III Stationary Source Specific Methods. EPA-600/4-7-027b, August 1977.
- 4. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019, March 1979.
- 5. Test Methods for evaluating Solid Waste, SW-846, 2<sup>nd</sup> edn., July 1982.
- 6. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," Federal Register, Vol 49, No. 209, October 26, 1984.

Section A
Appendices

A-1 Resumes of Key CompuChem Personnel

# ROBERT E. MEIERER Director Quality Assurance CompuChem® Laboratories

Responsibilities

Mr. Meierer is the Director Quality Assurance and is responsible for assuring that all CompuChem® laboratories consistently produce high quality and reliable data and that all necessary certification and licensing requirements are met by the laboratories.

Education

Mr. Meierer received an Associate degree in Industrial Chemistry from the Erie County Technical Institute and an undergraduate degree in Chemistry from the State University of New York at Buffalo. He has taken advanced studies in Analytical Chemistry and Business Administration from the State University at Buffalo.

Experience

In his previous position with CompuChem® as Technical Development Scientist, Mr. Meierer was responsible for providing technical assistance to operational laboratories for procedure development and implementation and problem solving. Mr. Meierer had previously been employed with CompuChem® as Manager Analytical Laboratories where he was responsible for directing the efforts of the Sample Preparation Laboratories, the Inorganics Laboratory, the GC Laboratory, and the Standards Laboratory:

Prior to joining CompuChem®, Mr. Meierer held positions as Laboratory Manager with Radian Corporation and as Department Head, Analytical Laboratory; Special Contaminants Monitoring, The Carborundum Company.

Through the variety of laboratory positions Mr. Meierer had held, he has gained 3 years of experience in the interpretation of mass spectra gathered in GC/MS analysis. Additionally, Mr. Meierer has gained 7 years experience in the preparation of extracts from environmental or hazardous waste samples. Further, he has gained 3 years experience in organochlorine pesticide residue and PCB analysis, including clean-up procedures such as column chromatography on environmental samples.

JEANA W. ASPREY
Junior Chemist
CompuChem® Laboratories

Responsibility

Ms. Asprey is responsible for performing sample analysis using ICP and flame/furnace AAS.

Education

Ms. Asprey received an undergraduate degree in Biology from the University of Tennessee. Additionally, she has pursued studies in Computer Science.

Experience

Prior to joining CompuChem, Ms. Asprey had over five years experience in technician and chemist functions. Specifically, Ms. Asprey's background includes operating and maintaining ICP, AA and IC instrumentation.

JOHN C. TZAVARAS
Senior Chemist
CompuChem® Laboratories

Responsibility

Mr. Tzavaras is employed as a Senior Chemist, with responsibility for operating the Inorganics Laboratory for the determination of metals, cyanides, and phenols.

Education

Mr. Tzavaras received his undergraduate degree in Chemistry from Tufts University.

Experience

Prior to joining CompuChem®, Mr. Tzavaras was employed at Instrumentation Laboratories, Inc., as Product Specialist where his duties included applications development and explaining methodology and instrumentation to customers. Mr. Tzavaras had previously been employed at Herbert V. Schuster Laboratories as a Chemist where he analyzed drug, food and environmental samples.

Through the various laboratory positions Mr. Tzavaras has held, he has gained three years of experience in the determination of metals, cyanides, and phenols.

A-2 Example Laboratory Report - CompuChem Laboratories, Inc.



April 8, 1987

Mr. Joe Doe CompuChem Labs. RTP, NC 27709

Dear Mr. Doe:

We at CompuChem® are pleased to provide our report for the analysis you requested. Data for the following sample are enclosed:

Your ID	Our ID	Analysis	Order	Description of Work	Report
Number	Number	Code	Number	Requested	Silver
xxxxx	xxxxx	642,045	00000	E.P. Toxicity Leachate Metals	xxxx

In this report we have included the analytical results, the method reference, and the quality control summary. If any anomalies were encountered in this analysis, they would be referenced in an attached Quality Assurance Notice(s). Instrument documentation is provided with reports purchased in our Gold Report format.

To obtain additional technical information concerning this report, please contact your Sales Representative. In addition to resolving your questions, they can provide you with a complete overview of our line of services and assist you in identifying those services which will effectively and efficiently support your monitoring program.

For your convenience, your Customer Service Representative can help you place a new order, obtain information about a sample's status or obtain assistance with sample logistics. Your Sales Representative and your Customer Service Representative can be reached at 1/919-549-8263.

Thank you for choosing CompuChem®. We would like to continue providing you analytical support and services. We would appreciate your comments regarding the quality of services you have received from CompuChem®; client satisfaction is important to us. Please address your comments to your Sales or Customer Service Representative at the address given below.

Sincerely,

Mary E. Mitchell Supervisor, Report Deliverables

cc: Accounting
 (Cover letter only)



# ANALYTICAL REPORT OF DATA SUBMITTED TO:

Mr. Joe Doe CompuChem Labs RTP, NC 27709

# CHRONICLE

NO.	IDENTIFIER	NUMBER	RECEIVED	PERFORMED	ANALYZED
ITEM	SAMPLE	COMPUCHEM®	DATE SAMPLE	DATE E.P. TOXICITY LEACHATE	DATE METALS

## COMPOUND LIST - INORGANICS PRIORITY POLLUTANTS

# SAMPLE IDENTIFIER: COMPUCHEM SAMPLE NUMBER:

		CONCENTRATION (MG/_)
2. B. 3. C. 4. Cl 5. Ll 6. M 7. S	RSENIC, TOTAL ARIUM, TOTAL ADMIUM, TOTAL HROMIUM, TOTAL EAD, TOTAL ERCURY, TOTAL ELENIUM, TOTAL ILVER, TOTAL	0.050 1.0 0.010 0.050 0.050 0.00020 0.010 0.050

- U Indicates element was analyzed for but not detected. Report with the detection limit value (e.g. 10U).
- Value If the result is a value greater than or equal to the instrument detection limit but less than the EPA Contract Laboratory Program (CLP) Contract Required Detection Limit (CRDL), the value is reported in brackets (i.e.,[10]).

A-3 Facilities and Equipment - CompuChem Laboratories, Inc.

LABORATORY FACILITIES, EQUIPMENT, AND SERVICE

#### 1 Introduction

CompuChem® Laboratories, Inc. is located in Research Triangle Park, N.C., 15 miles west of Raleigh. The laboratory facility has 32,800 square feet of office and laboratory space. The administrative facility, located in an adjacent building, has over 8,700 square feet of office space. Electrical power is supplied by Duke Power Company, with a service capacity of 2000 amperes at 480 volts. The environmental controls for the heating, ventilating, and air conditioning systems are Honeywell Electric and provide automatic starting and stopping as well as temperature control. Building security is controlled by a Rusco Electronic Card Entry Access System. The exterior doors as well as the doors of various controlled access areas within the building are equipped with electronic card readers. A burglar alarm system has been intergrated with the Rusco System to provide protection when the facility is closed. Smoke detectors, as well as associated pull stations and fire alarm horns, are provided throughout the building for fire protection. Adequate fire extinguishers and emergency equipment are also provided. All critical temperature areas such as refrigerators, freezers and computer rooms are monitored 24 hours a day by an off-site monitoring firm to ensure maintenance of proper tem peratures. The fire and burglar alarms are also monitored by this firm. When an alarm sounds, the off-site personnel alert the appropriate laboratory personnel, the Sheriff's office, or the Fire Department, as necessary.

CompuChem® Laboratories contains sophisticated, state-of-the art instrumentation and data processing equipment capable of performing most organic and inorganic

analyses. Two Hewlett-Packard Series 68 mainframe computers are dedicated to scheduling and tracking sample analysis through the laboratories and provide networking capabilities. An additional HP-3000 Series 39 minicomputer is dedicated to systems research. The computerized laboratory management system is accessed by laboratory, marketing, systems, and accounting personnel via more than 90 computer terminals.

The 23 GC/MS instruments are configured with both packed and capillary GC columns, and have accessories for purge and trap, direct injection, or solid probe for introduction of samples. Both electron impact and chemical ionization sources are available. Each GC/MS instrument is equipped with its own dedicated microprocessor for data processing.

The GC Laboratory's 18 gas chromatographs are equipped with autosamplers or Tecmar LSC-2 purge-and-trap devices. Available detectors include Flame Ionization (FID), Electron Capture Thermoionic Specific (also called NPD or AFID), Photoionization (PID), and Electroculometric (also called a Hall Detector) detectors.

The laboratory also has a full complement of support equipment and instrumentation, such as glove boxes and hoods, walk-in refrigerators, freezer units, autoanalyzers and sonicators. All instruments are maintained by five experts employed by CompuChem® on a full-time basis.

Instrument log books are maintained for each individual instrument in each of the laboratories (GC, GC/MS, Inorganics). Each laboratory instrument has a unique log book.

Security is maintained at all times. The visitors' entrance is secured, and visitors are escorted through the facility by members of the staff only after signing a Visitor's Register.

The following sections describe the laboratory area by function and equipment. It should be noted that the floor plan allows the efficient movement of samples between working areas.

# 6.2 <u>Laboratory Areas</u>

Shipping and Receiving: This area is located adjacent to the laboratory section of the building. Samples arriving are identified and introduced into the scheduling and control system. The sample receiving area for environmental samples has about 2,450 square feet of floor space. The receiving area has 102 square feet of bench space for receiving and opening samples, three data entry stations, one laboratory sink and ample storage shelving. A 2,500 cubic foot refrigerator  $(4^{\circ}C \pm 2^{\circ}C)$  is provided for storage of environmental samples.

<u>Walk-in Refrigeration System</u>: This area is accessed from the shipping and receiving area as well as from the central laboratory corridor. This unit has two independent refrigeration systems, is temperature controlled to  $4^{\circ}C + 2^{\circ}C$  and is equipped with an activated carbon air filtering system, which maintains an environment free of organic vapors. The temperature is recorded daily. Both entrances are secured by locks and the temperature-activated alarm system is tied into a private security service. In the event of unauthorized access or temperature flucuations, appropriate parties are notifed by the private security service.

Extractions and Preparations Laboratory: This area is equipped with hoods as well as extraction equipment sufficient to process many thousands of samples per month. The environmental sample preparation laboratory has 2,024 square feet of space, two 8' fume hoods, three IEC centrifuges, two vacuum ovens, two sinks, six water baths, and 220 square feet of bench space. The air handling system for the sample preparation laboratory was custom designed for

the extraction process. Conditioned 100% outdoor air is supplied into the room through linear diffusors and exhausted through exhaust ducts which extend from wall to wall on the north and south ends of the laboratory. This method maintains air flow at the workstations at all times and virtually makes the room a large walk-in fume hood. A complete air exchange occurs every two minutes. Separate exhausts are provided for furnaces and hoods. A low air flow alarm sounds if a condition develops causing low air flow into the room. Adequate cabinet space is provided. Specially-designed water baths controlled and programmable to temperature and duration are also used. The glassware preparation room has 750 square feet of floor space and is equipped with two glassware washers, 26 feet of stainless steel counters with four built-in sinks, and one 72 cubic foot annealing oven.

Solvent Storage Area: This area is accessible only through a secured door adjacent to the extraction and preparation area. The room is designed with reinforced concrete walls, alarm systems and a roof that relieves pressure in the event of an accident.

GC Laboratory: The laboratory's eighteen gas chromatographs are equipped with autosamplers or purge-and-trap devices and are interfaced with a Hewlett-Packard 1000 laboratory computer for data processing (all of which are installed on a raised computer floor). A variety of detectors are attached to the GCs.

GC/MS Laboratory: The special features included in this area are numerous. All twenty-three GC/MS systems are raised on a computer floor. This allows gas, water, cooling and exhaust systems required to support each

instrument to be introduced to the room independently, beneath the floor. Equipment is arranged in efficient working clusters. In this way, specific instruments can be utilized for specific types of analyses. For example, several instruments are totally dedicated to volatile organic analyses. These instruments are never subjected to semi-volatile work; therefore, cross-contamination of the instruments is eliminated. Furthermore, each cluster of instruments is staffed by experts familiar with the protocols associated with each specific procedure. This staffing system allows intimate daily interaction between the operator, his or her instruments and the methodologies required. All other instruments are dedicated in a similar fashion. Also located in a section of this area are two Hewlett Packard 3000 Computers used for support of scheduling and control activities and data networking. The combined GC/MS and Computer Room has a total of 3,380 square feet. Each GC/MS and computer is provided with an individual power supply from a breaker panel located within the lab. The GC/MS instruments are powered by three 1-phase, 75 KVA 480/220 volt isolation transformers. The computers are powered by one 3-phase 75KVA 480/208 volt isolation transformer. Helium, the carrier gas used, is supplied from a manifold system in an adjacent room through a piping system under the raised floor. There are three of these systems, each having a catalytic scurbber to remove traces of oxygen and water, prior to entering an instrument.

Standards Laboratory: This area is separated completely from all other laboratories and is equipped with its own GC instrument. Refrigeration, glove box and hood units are located in this area. The entrance to this area is secured by tow magnetic card locks and a cypher lock.

Inorganics Laboratory: This area is separated completely from all other laboratories and has one inductively Coupled Argon Plasma (ICAP) unit, one technicon autosampler, two Atomic Absorption Separated (AAS) instruments, and one UV/visible spectrophotometer. Several other analytical instruments required to perform classical analyses are also located in this laboratory. Hood systems are also an integral part of this laboratory.

Extract Storage: Sample extracts are stored in specially-designed refrigeration units located adjacent to the Extraction Laboratory. These refrigeration units are accessed on a limited basis by a sample custodian only. Entrance is on a "need only" basis and requires a key to gain entrance. These refrigeration units are also connected to an alarm system. In the event of temperature fluctuations outside acceptable levels (4°C  $\pm$  2°C), appropriate parties are notified by a private security service and the problem is corrected by laboratory staff.

High Hazard Laboratory: A limited access laboratory has been designed for sample preparation aspects associated with high-hazard samples. For example, all samples requiring analysis for 2,3,7,8-TCDD are prepared in this lab. Direct access to the laboratory is by means of a cypher lock. The hoods employed are equipped with a HEPA filtration unit. Laboratory personnel use more protective clothing than the other extraction laboratory personnel; i.e. full sack suits, booties, face masks, etc.

# FACILITY SPACE ALLOCATION

# TOTAL BUILDING SQUARE FEET 32,800

# INDIVIDUAL AREAS

1.	Sample Receiving	2,450 sq. ft.
2.	Glassware Prep	750 sq. ft.
3.	Extractions	2,024 sq. ft.
4.	High Hazard	468 sq. ft.
5.	GC/MS	3,200 sq. ft.
6.	Standards	312 sq. ft.
7.	Metals (Inorganics)	735 sq. ft.
8.	RIA Analysis	312 sq. ft.
9.	GC	775 sq. ft.
10.	Clinical Recv.	936 sq. ft.
11.	Solvent Storage	542 sq. ft.
12.	Utility	960 sq. ft.
13.	Walk-In Refrigerator	702 sq. ft.
14.	Walk-in Freezer	364 sq. ft.
15.	Miscellaneous (Canteen, Corridors, Rest Rooms, etc.)	5,000 sq. ft.
16.	Office	13,270 sq. ft.

# 6.3 Equipment

# GC Laboratory

	Make	Model
2	Perkin-Elmer	Sigma 3Bs
	Varian	3700
	Hewlett-Packard	5880
	Hewlett-Packard	5880
	Hewlett-Packard	5790
9	Varian	3400s

# Inorganics Laboratory Instrumentation

Make		Model
Jarrel-ASH ICP		1100
Instrumentation Laboratory		Video 22 aa/ae
Instrumentation Laboratory		Viedo 12 aa/ae
Instrumentation Laboratory		755 Control Temp Furnance
Instrumentation Laboratory		755 Control Temp Furnance
Varian		UV-Vis Spectrophotometer Cary 219
Technicon		Cyanide/Phenol Autoanalyzer
Orion Research		Microprocessor ionalyzer/901
Instrumentation Laboratory	•	Atomic Vapor Accessory 440
Fisher Mercury Analyzer		HG3

# GC/MS Laboratory Instrumentation

Make	Model
Finnigan	0WA-20
Finnigan	OWA-20
Finnigan	OWA-20
Finnigan	0WA-20
Finnigan	OWA-20
Finnigan	OWA-20
Finnigan	0WA-20
Finnigan	0WA-20
Finnigan	OWA-20
Finnigan	OWA-20
Finnigan	0WA-20
Finnigan	OWA-20B
Finnigan	OWA-20B
Finnigan	OWA-20B
Finnigan	OWA-1020 GC/MS
Finnigan	OWA-1020 GC/MS
Finnigan	OWA-1020 GC/MS
Finnigan	OWA

# A-4 EP Toxicity Test SOP

- 1. Analytical Method
- 2. Equations and Calculations

### SAMPLE PREPARATION PROCEDURE 801

# EP Toxicity Test Procedure

# A. Extraction Procedure (EP)

- A representative sample of the waste to be tested (minimum size 100 grams) should be obtained. {For detailed guidance on conducting the various aspects of the EP see "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," SW-846, U.S. Environmental Protection Agency Office of Solid Waste, Washington, D.C. 20460.¹} (In Library).
- 2. The sample should be separated into its component liquid and solid phases using the method described in "Separation Procedure" below. If the solid residue<sup>2</sup> obtained using this method totals less than 0.5% of the original weight of the waste, the residue can be discarded and the operator should treat the liquid phase as the extract and proceed immediately to Step 8.
- 3. The solid material obtained from the Separation Procedure should be evaluated for its particle size. If the solid material passes through a 9.5 mm (0.375 inch) standard sieve, the operator should proceed to Step 4. If the particle size is larger than specified above, the solid material should be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5 mm (0.375 inch) sieve.
- 4. The solid material obtained in Step 3 should be weighed and placed in an extractor with 16 times its weight of deionized water. Do not allow the material to dry prior to weighing. For purposes of this test, an acceptable extractor is one which will impart sufficient agitation to the mixture to not only prevent stratification of the sample and extraction fluid but also insure that all sample surfaces are continously brought into contact with well mixed extraction fluid.

Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

The percent solids is determined by drying the filter pad at 80° C until it reaches constant weight and then calculating the percent solids using the following equation:

(weight of pad + solid)
- (tare weight of pad)

- X 100 = % solids

Initial weight of sample

# Separation Procedure

Equipment: A filter holder, designed for filtration media having a nominal pore size of 0.45 micrometers and capable of applying a  $5.3~\rm kg/cm^2$  (75 psi) hydrostatic pressure to the solution being filtered shall be used. For mixtures containing nonabsorptive solids, where separation can be affected without imposing a  $5.3~\rm kg/cm^2$  pressure differential, vacuum filters employing a  $0.45~\rm micrometers$  filter media can be used. (For further guidance on filtration equipment or procedures see "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.")

# Procedure:3

- i. Following Millipore's directions, the filter unit should be assembled with a filter bed consisting of a 0.45 micrometer filter membrane. For difficult or slow to filter mixtures a prefilter bed consisting of the following prefilters in increasing pore size (0.65 micrometer membrane, fine glass fiber prefilter, and coarse glass fiber prefilter) can be used.
- ii. The waste should be poured into the filtration unit.
- iii. The reservoir should be slowly pressurized until liquid begins to flow from the filtrate outlet at which point the pressure in the filter should be immediately lowered to 10-15 psig. Filtration should be continued until liquid flow ceases.
- iv. The pressure should be increased stepwise in 10 psi increments to 75 psig and filtration continued until flow ceases or the pressurizing gas begins to exit from the filtrate outlet.
- v. The filter unit should be depressurized, the solid material removed and weighed and then transferred to the extraction apparatus, or, in the case of final filtration prior to analysis, discarded. Do not allow the material retained on the filter pad to dry prior to weighing.
- vi. The liquid phase should be stored at 4° C for subsequent.

This procedure is intended to result in separation of the "free" liquid portion of the waste from any solid matter having a particle size > 0.45 mm. If the sample will not filter, various other separation techniques can be used to aid in the filtration. As described above, pressure filtration is employed to speed up the filtration process. This does not alter the nature of the separation. If liquid does not separate during filtration, the waste can be centrifuged. If separation occurs during centrifugation the liquid portion (centrifugate) is filtered through the 0.45 mm filter prior to becoming mixed with the liquid portion of the waste obtained from the initial filtration. Any material that will not pass through the filter after centrifugation is considered a solid and is extracted.

- After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater that 5.0, the pH of the solution should be decreased to  $5.0 \pm 0.2$  by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and if the pH rises above 5.2, 0.5N acetic acid should be added to bring the pH down to  $5.0 \pm 0.2$ . However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hours and maintained at  $20^{\circ}$  - $40^{\circ}$  C ( $68^{\circ}$   $104^{\circ}$  F) during this time. The following manual procedure shall be employed:
  - a. A pH meter should be calibrated in accordance with the manufacturer's specifications.
  - b. The pH of the solution should be checked and, if necessary, 0.5N acetic acid should be manually added to the extractor until the pH reaches  $5.0\pm0.2$ . The pH of the solution should be adjusted at 15, 30 and 60 minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5N pH units.
  - c. The adjustment procedure should be continued for at least 6 hours.
  - d. If at the end of the 24-hour extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to  $5.0 \pm 0.2$  and the extraction continued for an additional four hours, during which the pH should be adjusted at one hour intervals.
- 6. At the end of the 24 hour extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

V = (20)(W)-16(W)-A

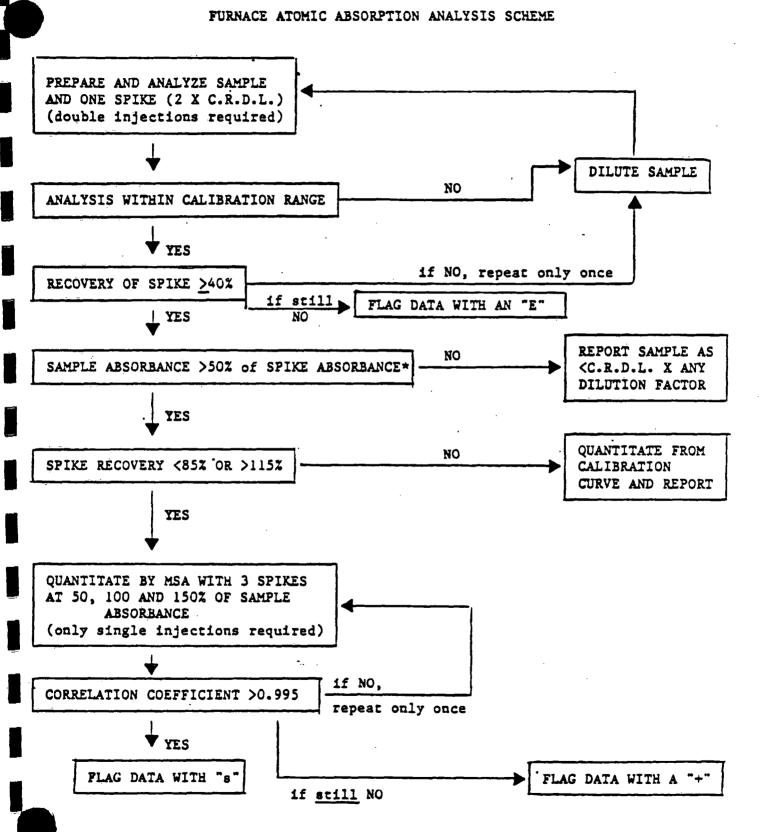
V = ml deionized water to be added

W = Weight in grams of solid charged to extractor

A = ml of 0.5N acetic acid added during extraction

- 7. The material in the extractor should be separated into its component liquid and solid phases as described under "Separation Procedure."
- 8. The liquids resulting from Steps 2 and 7 should be combined. This combined liquid (or the waste itself if it has less than ½ percent solids, as noted in Step 2) is the extract. Dilute liquid phase to 2.51. Fill 2 11 glass sample bottles and 1 500ml plastic sample bottle and label appropriately. Store at 4°C.

Figure 1.



spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample)

- 1. All furnace analyses, except during Full Methods of Standard Addition (MSA), will require duplicate injections for which the average absorbance or "concentration" will be reported. All analyses must fall within the calibration range. The raw data package must contain both absorbance or "concentration" values, the average value and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than CRDL, the duplicate injection readings must agree within 20% RSD or CV, or the sample must be rerun once. If the readings are still out, flag the value with an "M" on Form I.
- 2. All furnace analyses for each sample will require at least a single analytical spike to determine if the MSA will be required for quantitation. Analytical spikes are not required on the predigest spike samples. The spike\* will be required to be at a concentration (in the sample) twice the contract required detection limit (CRDL). The percent (IR) of the spike, calculated by the same formula as Spiked Sample analyses (Exhibit E), will then determine how the sample will be quantitated as follows:
  - a) If the spike recovery is less than 40%, the sample must be diluted and rerun with another spike. Dilute the sample by a factor of 5 to 10 and rerun. This step must only be performed once. If after the dilution the spike recovery is still <40%, report data and flag with an "E" to indicate interference problems.
  - b) If the spike recovery is greater than 40% and the sample absorbance or concentration is <50% of the spike<sup>+</sup>, report the sample as less than the CRDL or less than the CRDL times the dilution factor if the sample was diluted.
  - c) If the sample absorbance or concentration is >50% of the spike<sup>†</sup> and the spike recovery is between 85% and 115%, the sample should be quantitated directly from the calibration curve.
  - d) If the sample absorbance or concentration is >50% of the spike<sup>+</sup> and the spike recovery is less than 85% or greater than 115%, the sample must be quantitated by MSA.
- 3. The following procedures will be incorporated into MSA analyses.
  - a) Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

<sup>\*</sup>Spikes are post diges: spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the <u>digested sample</u>. The unspiked sample aliquot must be compensated for any volume change in the spike samples by addition of deionized water to the unspiked sample aliquot.

<sup>+&</sup>quot;Spike" is defined as (absorbance or concentration of spike sample) minus (absorbance or concentration of the sample).

excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.

- c) Spikes\* should be prepared such that:
  - Spike 1 is approximately 50% of the sample absorbance.
  - Spike 2 is approximately 100% of the sample absorbance.
  - Spike 3 is approximately 150% of the sample absorbance.
- d) The data for each MSA analysis should be clearly identified in the raw data documentation along with the slope, intercept and correlation coefficient (r) for the least squares fit of the data and the results reported on Form VIII. Reported values obtained by MSA are flagged on the data sheet (Form I) with the letter "s".
- e) If the correlation coefficient (r) for a particular analysis is less than 0.995 the MSA analyses must be repeated once. If the correlation coefficient is still <0.995, the results on Form I must be flagged with "+".

# 9. Laboratory Control Sample Analysis

Laboratory Control Sample (LCS) - Aqueous and solid laboratory quality control samples must be analyzed for each analyte using the same sample preparation and analytical methods employed for the EPA samples received.

The aqueous LCS solution must be obtained from EPA (if unavailable, the EPA Initial Calibration Verification solution may be used). The aqueous LCS must be prepared and analyzed with the samples for each of the procedures applied to each case of samples received. One aqueous LCS must be analyzed for every 20 samples received, or for each batch of samples digested whichever is more frequent (see Exhibit D). Each data package must contain the results of all the LCS analyses associated with the samples on that Case. For cyanide, the distilled mid-range calibration standard may be used as the aqueous LCS (see Section 8.3.2.1, Exhibit D). An aqueous LCS is not required for mercury analysis.

<sup>\*</sup>Spikes are post digest spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the <u>digested sample</u>. The unspiked sample aliquot must be compensated for any volume change in the spiked samples by addition of deionized water to the unspiked sample aliquot.

<sup>+</sup>A group of samples prepared at the same time.

All aqueous LCS results will be reported on Form VII in terms of true concentration and percent recovery (%R) as calculated by:

#### ZR = (Observed/True) x 100

where "observed" is the measured concentration. If the Z recovery for the aqueous LCS falls outside the control limits of 80% - 120%, the analyses must be terminated, the problems corrected and the previous samples associated with that LCS re-analyzed (ie., previous 19 samples or the batch of samples from the case).

Once a month, a solid LCS, available from EMSL-LV must be prepared and analyzed using each of the procedures applied to the solid samples received. If this EPA solid LCS is unavailable, other EPA Quality Assurance Check samples or other certified materials may be used.

The monthly results of the solid LCS samples should be reported on a duplicate Form VII and submitted monthly to EMSL/Las Vegas and SMO on the 15th of every month.

If the percent recovery for the solid LCS sample is outside the control limits established by EPA, no further sample analyses may be done until the analytical problems are solved, and satisfactory LCS results are obtained.

A-5 Resumes of Key Metcalf & Eddy Personnel

#### BRUCE E. GOODWIN

#### **EDUCATION:**

BS, Chemistry, Tufts University, 1970 MS, Chemistry, Tufts University, 1973

#### PROFESSIONAL MEMBERSHIPS:

American Chemical Society

#### GENERAL BACKGROUND:

Mr. Goodwin is a Technical Specialist in Metcalf & Eddy's Chemical Waste Management Division and serves as an advisor in analytical and environmental chemistry. Mr. Goodwin is responsible for the selection and management of subcontractor analytical testing services and the formal development of QA/QC procedures for environmental monitoring. He has participated in patent infringement and other litigation; chemical process development and failure diagnosis; environmental studies including literature review, chemical fate and transport studies, field sampling, and chemical analysis; hazardous waste treatment including process design, feasibility studies, and contamination assessment; industrial hygiene monitoring; and basic and applied research and development.

#### **EXPERIENCE:**

- . Provided technical direction of research and development programs, personnel supervision, contract administration including budgeting and scheduling, technical report writing, and sales.
- . Provided several studies of land treatment technology for the U.S. EPA, assisting with the drafting of hazardous waste land treatment regulations.
- . Performed studies for detailed field surveys of six industrial land treatment sites located across the country. Reviewed process and waste stream chemistry, economics, waste disposal procedures, management practices, field sampling and chemical analysis of wastes, soils, and vegetation in order to understand the environmental fate of various waste stream constituents.
- . Assessed the feasibility of land treatment as an alternative means of waste disposal for a specific industrial waste, which involved review of process and waste stream chemistry and study of available disposal sites.

### B. E. Goodwin (Continued)

- . Investigated the statistical requirements for monitoring of land treatment sites. Studied sampling plans, hypothesis testing, sample size considerations, and statistical techniques for data treatment in the design of monitoring programs.
- Served as a member of a team responsible for process control and monitoring of a full-scale dual-alkali flue gas desulfurization system for the Southern Power Company in Chattanoochle, Florida, through a one-year EPA-funded study which involved operation and monitoring of the system.
- . Assisted a major U.S. household consumer products manufacturer with problems encountered during pilot production and test marketing of a new product. Reviewed process equipment and conditions, identified the specific factors responsible for the observed problems, and recommended quality control procedures to avoid recurrence of similar problems.
- . Assisted in an investigation of an explosion in a dielectric heating unit in a bakery owned and operated by a major U.S. food products company. Collected and evaluated evidence from the failed and undamaged units and the tested various hypotheses concerning the cause of the explosion.
- . Worked with management and technical staffs of a small manufacturing company to determine the nature and extent of environmental contamination resulting from previous wastewater disposal practices. Acted as technical representative of the client in meetings with state and local regulatory authorities. Reviewed manufacturing and process data to determine the nature of any contamination and to recommend specific species as indicators of the presence or absence of contamination in environmental samples. Designed and implemented sampling and technical analysis programs for surface waters, groundwater, and soils on and near the client's property. Identified and reviewed the technical feasbility and costs of possible remedial actions. Reported findings in discussions, presentations, letters, and technical reports prepared for submission to state and local regulatory authorities.
- Developed and applied wet chemical and instrumental methods of analysis to high purity metals and alloys, inorganic compounds, and various industrial effluents for the Ventron Corporation in Massachusetts.
- . Supervised fourteen technicians providing chemical analytical support to a R&D department at Uniroyal Chemical in Connecticut.

#### PUBLICATIONS:

. "Literature-Review Screening Techniques for the Evaluation of Land Treatment of Industrial Wastes," Berkowitz, J.B., B.E. Goodwin, J.C. Harris, and K. Scow, NTIS Report No. PB 110 386, November 1983.

## B. E. Goodwin (Continued)

- Land Treatment Field Studies, Berkowitz, J.B., S.E. Bysshe, B.E. Goodwin, J.C. Harris, D.B. Land, G. Leonardos, and S.L. Johnson, NTIS Report No. PB 83 241 265, September 1983.
- . <u>Surface Sampling Techniques</u>. Goodwin, B.E., J.R. Aronson, R.P. O'Neil, M.A. Randel, and E.M. Smith, Report DRXTH-TR-CR-82179, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland, September 1982.
- Evaluation of Seasonal/Soil Groundwater Pollutant Pathways via SESOIL. Bonazountas, M., J.M. Wagner, and B.E. Goodwin. Final Draft Report, U.S. EPA Contract 68-01-5949, Task 9. U.S. EPA, Washington, DC, 1982.
- . State of Knowledge," Harris, J.C., R.C. Anderson, B.E. Goodwin, and C.E. Rechsteiner. In <u>Study of State-of-the-Art of Dioxin from Combustion Sources</u>, The American Society of Mechanical Engineers, New York, 1981.
- . An Exposure and Risk Assessment for Nickel, McNamara, P.W., M. Byrne, B. Goodwin, K. Scow, W. Steber, R. Thomas, and M. Wood. Final Draft Report, U.S. EPA Contract 68-01-5949, Task 3, U.S. EPA, Washington, DC, December 1981.
- . "Statistical Analysis of Trace Metal Concentrations in Soils at Selected Land Treatment Sites," Grossman, M.A., B.E. Goodwin, and P.M. Brenner. Proceedings of the Seventh Annual Research Symposium on Land Disposal: Hazardous Waste. EPA-600/9-81-002b, March 1981.
- . "Identification of Hazardous Waste for Land Treatment Research," Berkowitz, J.B., J.C. Harris, and B.E. Goodwin. Proceedings of the Seventh Annual Research Symposium on Land Disposal: Hazardous Waste. EPA-600/9-81-02b, March 1981.
- "Generation of Test Atmospheres of Toxic Substances for Evaluation of Air Sampling Methods," Anderson, R.C., E.C. Gunderson, D.M. Coulson, B.E. Goodwin, and K.T. Menzies. In <u>Analytical Techniques in Occupational Health Chemistry</u>, ACS Symposium Series 120, American Chemical Society, Washington, DC, 1980.
- . "Field Verification of Land Cultivation Practices, "Berkowitz, J.B., S.E. Bysshe, B.E. Goodwin, J.C. Harris, D.B. Land, G. Leonardos, and S. Johnson. Paper presented at Sixth Annual Research Symposium, Treatment and Disposal of Hazardous Waste, Chicago, IL, March 1980.
- . "Analysis of the Organic Portion of Landfarmed Industrial Wastes and Soils," Harris, J.C., J.B. Berkowitz, and B.E. Goodwin. Paper presented at AICHE 87th National Meeting, Boston, Massachusetts, August 1979.

## B. E. Goodwin (Continued)

. Methods for the Deterimination of Phosphoric Acid, PC13, PC15 and PyS10 in Air, Goodwin, B.E., J.C. Harris, and J.E. Oberholtzer, Final Report to the National Institute for Occupational Safety and Health, Contract No. 210-75-0038, NIOSH, Cincinnati, Ohio, May 1977.

#### Alan C. Ford

Company:

Metcalf & Eddy, Inc.

Title: Education:

Senior Engineering Technician, T-3 AS, Environmental Sciences, 1977

Mr. Ford is the Senior Engineering Technician in Metcalf & Eddy's Chemical Waste Management Division. His responsibilities include the development of sampling and analysis plans in support of inhouse engineering studies, assistance with the preparation of project specific quality assurance project plans, estimation of cost and manpower needs for pilot studies and field investigations for both inhouse and commercial laboratory clients, design and layout of laboratory spaces for clients constructing new facilities, and technical support to Metcalf & Eddy's inhouse analytical laboratories.

### Past Experience

Prior to joining the Chemical Waste Management Division, Mr. Ford was Supervisor of Metcalf & Eddy's Laboratory facility for four years. During this time his responsibilities included administration, purchasing, subcontracting and technical support as well as close supervision of the laboratory chemists. Time management, scheduling of projects, budgeting, written reporting of data to clients, assistance with design of bench-scale pilot studies and field sampling programs as well as preparation of commercial client proposals and quotations were routine job responsibilities.

#### On Site Piloting and Field Work

Mr. Ford is routinely involved during the execution of complex on-site sampling and analysis programs. During his 8 1/2 years with Metcalf & Eddy he has conducted the operation and monitoring of a pilot facility to investigate ozone disinfection of wastewater under stringent chemical and bacteriological standards; operated a 3 gpm pilot plant designed to evaluate six different types of granular activated carbon providing data for a confidential client interested in color removal at a dye manufacturing facility; set-up and operated two continuous-feed biological wastewater pilot plants involving single stage nitrification, septage treatment, metals and cyanide removal; provided field and laboratory support for a pilot plant investigating pulsator clarification; designed and operated a settling column study to determine the settleability of domestic waste in a marine environment; and prepared and implemented a sludge composting sampling and analysis program involving the design and support of field laboratories.

Mr. Ford routinely supervises the field staff as well as participates in the actual sampling of a variety of surface impoundments, streams, monitoring wells, sumps, pits, leachate collection systems, sewerage systems, sludge dewatering facilities and industrial pretreatment plants, as well as waste piles and containments of ash, dust, solid waste and soils. In addition he has successfully completed a 40 hour Health and Safety Training course approved for use by the U.S. Environmental Protection Agency.